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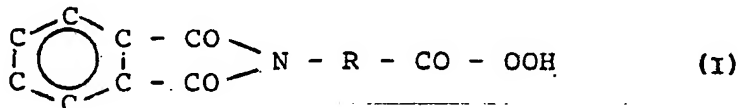
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(54) Title: PEROXYCARBOXYLIC ACIDS



(57) Abstract

The incorporation of many percarboxylic acids in household formulations is precluded from by adverse physicochemical properties, such as a propensity to decompose too quickly during storage or explode on impact or when subjected to pressure, though otherwise they would be effective disinfectants and bleaching agents, especially at hand-hot temperatures. The invention provides a stable and effective selected organic percarboxylic acid which satisfies general formula (1). in which R represents a pentamethylene group. The invention also provides bleaching, washing and disinfecting compositions containing the peracid and processes using it for such purposes.

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### Peroxycarboxylic acids

The present invention relates to peroxycarboxylic acids and more particularly to peroxycarboxylic acids which contain within their structure an imido linkage, to the  
5 preparation of such percarboxylic acids and to their use in bleaching compositions and in washing compositions.

Organic peroxycarboxylic acids, sometimes alternatively called percarboxylic acids or organic peracids, as a class, are potentially very useful oxidising agents as a result of  
10 their high electropotential which enables them to bleach very effectively a wide range of stains that mark domestic laundry or non-absorbent surfaces in the home and very useful disinfectants or sanitizers on account of their biocidal activity against a broad spectrum of pathogenic  
15 micro-organisms. Self-evidently, some percarboxylic acids are more effective than others in such activities, but the relative efficacy of the percompounds is only one key factor in determining the potential usefulness of such  
percompounds because they vary also in a second key area,  
20 which is the physical characteristics of the percompounds and specifically their sensitivity to impact, pressure, friction or thermal shock and their propensity to decompose during storage, either by themselves or promoted by contact with  
other components of washing or bleaching compositions and/or  
25 the rest of their environment. Variation in respect of both factors occurs as a direct result of what else is present in the percarboxylic acid molecule and the structural relationship of for example the various substituents to the percarboxylic acid group and to each other.

30 It is relatively easy for the skilled person in this

field to set out a number of desiderata, ie criteria he would want met for a peroxyacid to be an acceptable or very acceptable bleaching component, in practice. These criteria include not only effective washing and bleaching performance, but also sufficient resistance to impact, friction, pressure and thermal shocks to enable the compound by itself or in a formulation to be handled, transported and formulated, and an adequately long shelf-storage life, ie successful, safe and stable.

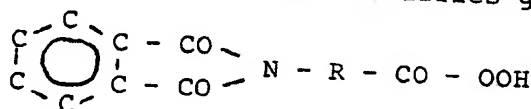
The impression may have been fostered that all peroxyacids are similar, as a result of the inclusion of a wide general formula for peracids in many patent specifications, eg US-A-4 259 201 of  $\text{HO-O-(CO)-R-Y}$  which appear to equate aliphatic and aromatic peroxyacids containing a wide range of substituents. Indeed, they do share, in general, a capability of bleaching domestic stains at lower molar amounts and at lower temperatures than hydrogen peroxide or persalts from which they are usually derived. However, various sub-classes of peroxyacids show considerable variation in the extent to which they enjoy enhanced bleaching properties and/or suffer from the tendency to decompose by shock and/or in storage. Thus, the task facing the skilled person in this field can be expressed simply; he is striving simultaneously to promote reactivity that improves bleach performance whilst suppressing reactivity that results in decomposition.

One of the sub-classes of peroxyacids tested in the course of the present investigations comprised those containing within their structure an imide link. As also happens with other peroxyacids, it was found that the physical and chemical properties of the imidoperoxyacids varied as the other substituent moieties were varied. It has been found that a selection from the imido-peracids can be made which simultaneously combines the chemico-physical properties of good storage stability and safe handling with good bleaching and disinfection performance.

In August 1989, ie between the dates of the priority and

present applications filed in respect of the instant invention, two applications by Ausimont S.r.l. were published, namely EP-A-0 325 288 and EP-A-0 325 289 which also describe a number of imidoperoxyacids. It can be deduced from the absence of data and the apparently indiscriminate breadth of their class that Ausimont did not investigate whether the physical characteristics of the peroxyacids accorded with those of a practical bleach and, accordingly, were also unaware of how the members of their class of peroxyacids differ from each other. Moreover, even as regards the performance of the compounds as bleaches, the essential point of '289, the specifications identify only imidoperoxyacids that are inferior bleaches compared with the present invention when measured against the real world stains of domestic laundry.

According to the present invention there is provided an organic peroxyacid which satisfies general formula (1):-



in which R represents a pentamethylene group.

In making the selection of the imido-peroxyacid of the instant invention, it is of practical importance to choose a complementary alkylene group containing an appropriate number of carbon atoms. This means choosing an alkylene group that is not too large or too branched, because the thermal stability tends to become worse as the size of the alkylene group increases and as the group deviates from linearity. There is progressive impairment of the storage stability of the peroxyacid as its constituent number of linear methylene groups is increased beyond the optimum. The presence of 5 linear methylene groups retains very good storage stability. On the other hand, it will also be recognised that the group must retain at least a minimum number of linear carbons between the imido nitrogen atom and the percarboxylic acid carbon atom. Where the separation is too short, the intrinsic safety of the compound is impaired.



In consequence, it will be recognised that imidoperoxyacids derived from natural aminoacids are generally unsuitable, because the two groups are separated by a single carbon. The practical selection of the peroxyacid compounds must also take into account their relative performance for example in bleaching, against the various types of stains encountered in domestic laundry. Thus, compounds which may enjoy acceptable safety and/or safety as when R contains 3 methylene groups may not be selected on performance grounds. The invention peroxyacid in which R represents a pentamethylene group is exceedingly attractive, not only as a result of its inherently attractive properties and performance, but also because the precursor imido-carboxylic acid can be obtained by condensation between an aromatic-1,2-anhydride and caprolactam, both of which are readily available starting materials in commercial amounts. Alternatively the 6-aminocaproic acid derivative of caprolactam can be used in the condensation reaction.

When considering selections of combinations of the peroxyacid substituents, it is convenient to pay attention to the melting point of purified peroxyacid. As a general guidance, for imidoperalkanoic acids, it is desirable to select compounds which have an apparent melting point at a temperature in excess of about 80°C. For many peroxyacids, this temperature more probably represents the onset of self-accelerating decomposition rather than a simple melting point.

The invention peroxycarboxylic acid can be made by reaction between the corresponding carboxylic acid and hydrogen peroxide in a strong mineral acid or organic acid reaction medium at a reaction temperature of below about 50°C, preferably from 5 to 30°C maintained until peroxyacid product precipitates out of solution, and thereafter separating the product from the reaction medium.

Similar reaction procedures are known for making poorly soluble aliphatic peroxyacids, and these can be applied to the manufacture of the invention peroxyacid. In effect,

the teaching in such prior publications as Siegel, et al in JOC, vol 27 pp1336-42 in 1961 entitled peroxides IX. New Method for the Direct Preparation of Aromatic and Aliphatic Peroxyacids can be employed, but modified as to the carboxylic acid starting material. Likewise, various processes described for the production of aliphatic peroxyacids in each of USP 2 813 896 (Krimm) USP 4 119 660 (Hutchins), USP 4 172 086 (Berkowitz), USP 4 233 235 (Camden), and USP 4 337 213 (Marynowski). Thus, the reaction medium for the peroxidation reaction when organic, is especially suitably an organic sulphonic acid, such as specifically methane sulphonic acid, which is probably the most readily available lower alkane sulphonic acid. When the reaction medium is a mineral acid, it is most preferably sulphuric acid or can desirably be phosphoric acid. Mixtures of the strong acids can be employed if desired.

It will also be recognised that where the reaction medium comprises a mineral acid, such as sulphuric acid, all or part of it can be premixed with the hydrogen peroxide to form an equilibrium mixture containing for example permonosulphuric acid that can itself perform the peroxidation reaction. Such premixing is beneficial because it separates the exothermic dilution/reaction between hydrogen peroxide and sulphuric acid from the peroxidation reaction, thereby enabling both to be controlled more readily and safely.

The attention of any readers not skilled in the art of peroxygen chemistry is directed to the potentially hazardous nature of peroxidation reactions and their products, to the need to take appropriate safety precautions at all times and to control the reaction conditions so as to ensure that the reaction mixture never at any time exceeds its SADT, self accelerating decomposition temperature and to carry out any initial tests on a very small scale.

Notwithstanding the above general warning which is of particular relevance to many peroxyacids, when they have been isolated, the peroxyacid of the instant invention is

characterised by its generally benign properties, specifically its relatively high stability and resistance to decomposition which it combines with highly acceptable bleach performance.

The imido-containing carboxylic acid, if not readily available, can be obtained by a conventional condensation between a suitably substituted aromatic-1,2-anhydride and the corresponding aminoalkanoic acid and especially conveniently, by reaction between an epsilon caprolactam and the aromatic-1,2-anhydride, phthalic anhydride. Conditions for this latter reaction route are described in Journal of American Chemical Society vol 70, 2115, (1948) in a paper by R E Benson and T L Cairns.

Whilst the instant invention relates primarily to the peroxyacid itself, it will be understood that it is possible to form magnesium salt derivative of the peroxyacid by neutralisation using magnesium oxide or similar compounds in media rendered alkaline to above the  $pK_a$  of the peroxyacid and recovery of the product that is permitted or induced to precipitate out. This corresponding salt shares the stability and performance of the peroxyacid itself and accordingly could be employed. However, it will be recognised that it is of benefit for washing and bleaching purposes to avoid the un-necessary introduction of cations that directly contribute to water-hardness, such as magnesium. This particular benefit is inherent in the use of the instant selection in acid form, but is not retained when the corresponding magnesium salt is used. Such magnesium salts tend to enjoy a markedly higher solubility and rate of dissolution than the acid form from which they are derived. Thus, it will be immediately recognised that where it is beneficial to employ a peroxyacid having high water solubility, the user can employ the magnesium salt form of the invention peroxyacid. Where it is desirable to employ peroxyacids having relatively low water solubility, so as to minimise or eliminate bleach spotting problems, the acid form of the invention is preferably used.

Hereinafter, unless the context clearly demands otherwise, a reference to the use of an invention peroxyacid or compositions containing it includes a reference to use of the corresponding magnesium salt.

The percarboxylic acid according to the instant invention is a particulate solid and can be employed by itself or can be incorporated as an active bleach component in bleaching or washing compositions containing a range of other ingredients, the selection and amounts of which are at the discretion of the formulator and determine the name for the compositions.

For bleach or bleach additive compositions, the peroxyacid normally comprises from 1 to 80%, and often from 5 to 50%, all %s herein being w/w of the respective composition unless otherwise stated. The remainder, 99 to 20%, comprises a diluent either by itself or together with a minor amount, such as up to 20% in total of optional components such as peroxygen stabilisers, surfactants, etc as indicated subsequently herein. The skilled reader will recognise that many of the diluents described herein as being suitable have hitherto been described as one or other of desensitising diluents or stabilising diluents or exotherm control agents in conjunction with named prior art organic peroxyacids such as DPDDA. Whilst the presence of such diluent compounds may have been necessary to perform that function for those prior art peroxyacids, it is a significant feature of the invention peroxyacid that the presence of the same diluents is optional and in practice their selection can be based upon any other desirable feature of those compounds, such as their cheapness or their advantageous washing or detergent-enhancing properties.

The diluent is often a salt selected from anhydrous or hydrated alkali or alkaline earth metal salts of halogen-free acids, and particularly of mineral acids, including salts of sulphuric, and ortho, pyro or hexa-meta phosphoric acids. Preferably, the metal is selected from sodium, potassium and magnesium and in many instances is sodium.

Hydrated, partially hydrated or anhydrous sodium sulphate is often chosen in view of its widespread availability, its properties and its cost. It will be recognised, though, that use of a phosphate salt may be preferred in view of its known capabilities of acting as a detergent builder, which can complement especially an unbuilt washing composition.

Other inorganic compounds that are suitable for use as diluents include ortho and meta boric acid and alkali metal salts thereof, and especially sodium salts. Such compounds can buffer solutions of the bleach or additive composition to a pH in the immediate region of the  $pK_a$  of the peroxyacid and consequently optimise bleach activity. The boric acids have also been used as exotherm control agents in compositions containing peroxyacids such as DPDDA that need to be protected against a tendency to decompose in an otherwise uncontrollable fashion if allowed to reach a quite low threshold temperature, but that property is unnecessary in conjunction with the invention peroxyacids on account of the safe nature of these selected imido peroxyacids.

Other suitable inorganic diluents include alkali metal carbonates/ bicarbonates, aluminium salts of the above-identified mineral acids, and natural or synthetic aluminosilicates and clays, such as zeolites A, X and Y, often in the sodium form, or swelling clays like bentonite. It will be clearly recognised that many of these diluents also enjoy the status of builders in washing compositions, and that each accordingly can perform its known functions such as hardness removal or peptising when employed in bleach compositions. When the bleach composition is intended as a scour, at least a proportion of the diluent and preferably at least half of the diluent comprises abrasive powdered materials, including silica, quartz, marble dust or kieselguhr.

A further and rather different class of suitable inorganic diluents comprises alkali metal or alkaline earth metal halides, especially chlorides and/or bromides and particularly sodium chloride, or sodium bromide or a

mixture of the two. By employing this class of diluents as at least a part of the diluents, the composition can generate in solution during use of the composition a halide such as chlorine or bromine which can complement the bleaching/sanitising effect of the invention imido peroxyacid.

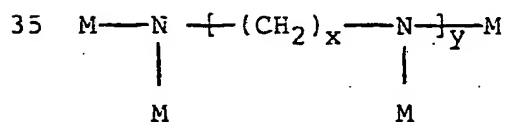
The diluent can comprise a hydrogen peroxide - developing solid persalts, or an inorganic persulphate, preferably in an amount of not more than 50% w/w of the composition. The term "persalt" herein relates primarily to alkali metal perborates, percarbonates and perphosphates, and especially the sodium salts, which generate hydrogen peroxide or the  $\text{HOO}^-$  anion depending on the solution pH, in situ and includes other hydrogen peroxide adducts which can do likewise. Preferred persalts include sodium perborate monohydrate or tetrahydrate and sodium percarbonate. Persalts include adducts with urea and related compounds, adducts with certain aluminosilicates and addition compounds with alkali/alkaline earth metal sulphate/chlorides in specified ratios. It will be recognised that the use of persalts as diluent, such as in at least 10% of the composition, enables the composition to be effective throughout a range of temperatures from ambient up to about 100°C.

In one more specialised type of bleaching compositions, namely effervescent composition, which are often intended primarily for cleansing dentures, but which can also be employed for many other purposes, the diluent for the invention peroxyacids preferably contains a gas generating system and if necessary a pH regulator. Compounds that are suitable for gas generating systems and as pH regulators are well known in conjunction with existing peroxyacids, and are described in EP-A-0 133 354 in the name of Interlox Chemicals Limited. The gas generating system often provides from 10 to 50% and comprises either a carbon dioxide generating combination of an alkali metal carbonate or bicarbonate with a solid water-soluble acid, and especially an organic acid

selected from tartaric, citric, lactic, succinic, glutaric, maleic, fumaric and malonic acids, preferably in an equivalent mole ratio of from 1.5:1 to 1:1.5 and especially at about 1:1, or an oxygen-generating compound such as anhydrous sodium perborate. The pH regulator often comprises 5 to 40% of the composition. To provide acidic conditions, it can comprise one or more of the aforementioned organic acids in an appropriate excess amount, or sulphamic acid or alkali metal bisulphates, and to provide alkaline conditions, it can comprise alkali metal silicates or excess carbonate/ bicarbonates. Selection of the percarboxylic salt form can be advantageous in such compositions.

In the main, the foregoing diluents have been inorganic. However, the invention peroxyacid can be diluted, if desired, with a range of organic substances, including hydrocarbon waxes, alkyl C1 to C6 esters of aromatic mono or di carboxylic acids, solid starches, gelatines and dextrans.

The bleach compositions can also contain, as indicated before, minor components such as peroxyacid stabilisers. The breadth of compounds suitable for this purpose is well-known in this art. These are often organic chelating compounds that sequester metal ions in solution, particularly most transition metal ions, which would promote decomposition of any peroxygen compounds therein, and many suitable ones being classified in the literature as carboxylic acid, hydroxycarboxylic or aminocarboxylic acid complexing agents or as organic amino- or hydroxy-polyphosphonic acid complexing agents, either in acid or soluble salt forms. Representative stabilisers expressed in acid form include picolinic acid, dipicolinic acid, quinolinic acid, gluconic acid, hydroxyethylene di phosphonic acid, and any compound satisfying the general formula:



in which M represents either  $-\text{CH}_2--\text{CO}_2\text{H}$  or  $-\text{CH}_2--\text{PO}_3\text{H}$ , x represents an integer selected from 1 to 6, and preferably is 2, and y represents an integer selected from 0, 1, 2 or 3. Within this general formula especially preferred

5 stabilisers include ethylenediamine tetra acetic acid (EDTA), ethylenediamine tetrakis (methylenephosphonic acid) (EDTMP), and diethylenetriamine pentakis (methylenephosphonic acid) (DTPMP). The amount of stabiliser is often up to 5% of the composition and in many instances is selected in the  
10 range of from 0.05 to 1%.

If present at all, a surfactant is present in bleaching compositions only in a small amount, such as up to about 5% and in many instances from 0.1 to 2% of the composition. It can be selected from the surfactants described subsequently  
15 herein for washing compositions.

The invention bleaching compositions will often comprise particulate mixtures, which can be stored loosely in conventional waxed boxes, or alternatively be enclosed in rupturable pouches or in porous or perforated bags or sacs  
20 through which bleaching solution can penetrate. Such mixtures can be obtained by dry blending the particulate components, or they can be aggregated using conventional agglomeration or granulation techniques, using water or a removable solvent and optionally a granulating aid hitherto  
25 described for use with an organic peroxyacid.

Alternatively, by virtue of their demonstrated ability to withstand pressure, all but the least resistant invention peroxyacids can be compressed in tablets and like bodies. Accordingly, it is possible to provide peroxyacids in easy  
30 to use predetermined dosage levels for the end user.

The bleaching compositions can be used by themselves, such as in a pre-wash bleach or a post-wash rinsing stage of a multistage laundry process or in cleansing both absorbent or non-absorbent (sometimes called "hard") surfaces. They  
35 are more usually employed in conjunction with a washing composition based upon surfactants. Naturally, surfactants and optional ingredients of washing compositions can be



premixed with the instant bleaching compositions to form bleach-containing washing compositions.

Washing compositions according to this further aspect of the present invention contain from 0.5 to 50% of the invention  
5 imido peroxyacids, from 1 to 90% surfactant, from 0 to 90% detergent builder, from 0 to 90% diluent and from 0 to 20% minor components. It will be recognised that the composition of the invention washing compositions range within very broad limits. Choice of the peroxyacid in acid  
10 form can be beneficial herein, in order to minimise or avoid spotting problems that can occur if excessive local concentrations of active bleach should be allowed to remain in contact with a dyed fabric for too long.

In many preferred compositions according to the present  
15 invention, one or more of the composition components are selected within the following narrower bands:-

imido peroxyacid	- 1 to 25%, particularly 2 to 10%
surfactant	- 2 to 40%, particularly 5 to 25%
builder	- 1 to 60%, particularly 5 to 40%
20 diluent	- 1 to 70%, particularly 5 to 50%
minor components	- 1 to 10% in total.

The surfactants for incorporation in solid compositions of the present invention can be selected from particulate or flaky anionic, cationic, non-ionic, zwitterionic, amphoteric  
25 and ampholytic surfactants and can be either natural soaps or synthetic. A number of suitable surfactants are described in chapter 2 of Synthetic Detergents by A Davidsohn and B M Milwidsky (6th edition) published in 1978 by George Godwin Ltd and John Wiley & Sons, incorporated herein by  
30 reference. Without limiting to these surfactants, representative sub-classes of anionic surfactants are carboxylic acid soaps, alkyl aryl sulphonates, olefin sulphonates, linear alkane sulphonates, hydroxy-alkane sulphonates, long chain and OXO alcohol sulphates, sulphated  
35 glycerides, sulphated ethers, sulpho-succinates, alkane sulphonates, phosphate esters, sucrose esters and anionic fluorosurfactants; representative classes of cationic

surfactants include quaternary ammonium or quaternary pyridinium salts containing at least one hydrophobic alkyl or aralkyl group, representative classes of nonionic surfactants include condensates of a long chain alkanol with  
5 either polyethylene oxides or with phenols, or condensates of long chain carboxylic acids or amines or amides with polyethylene oxide, and related compounds in which the long chain moiety is condensed with an aliphatic polypol such as sorbitol or condensation products of ethylene and propylene  
10 oxides or fatty acid alkanolamides and fatty acid amine oxides; representative classes of amphoteric/zwitterionic surfactants include sulphonium and phosphonium surfactants, optionally substituted by an anionic solubilising group. The proportion of surfactant, expressed as a fraction of all  
15 the surfactant present is often from 2/10 to 8/10ths anionic, from 0 to 6/10ths nonionic, and from 0 to 3/10ths for the other surfactants.

It will be recognised by the knowledgeable reader that many of the classes of diluent described herein above for  
20 use in bleaching compositions are also called detergent builders. These include specifically alkali metal phosphates, particularly tripolyphosphate but also tetrapyrophosphate and hexametaphosphate, especially the sodium salt of each, alkali metal, preferably, sodium  
25 carbonate, alkali metal, preferably, sodium borates, and the zeolites A, X and Y and clays like bentonite. Amongst organic compounds, the chelating compounds which were described herein as peroxygen stabilisers can also function as detergent builders. Particularly preferred chelating  
30 builders include nitrilotrisodium trisacetate (NTA), EDTA, EDTMP and DTPMP. Such chelating builders can be employed in a relatively small amount as an augmenting builder and peroxygen stabiliser, such as of 1 to 10% , or in  
cooperative partnership of equals in conjunction with a  
35 phosphatic or zeolitic or clay builder, the weight ratio of chelating to inorganic builders often being from 4:1 to 1:4, or alternatively they can be employed as the principal

builder in amounts of up to 40% such as in the range of 5 to 30% of the washing composition.

The other types of compounds that have been indicated to be suitable for use as diluents in a bleaching composition, can also be employed for the same primary purpose and secondary purpose, if any, in washing compositions, although it will be recognised that the presence of an effervescent system in washing compositions is comparatively rare. For the avoidance of doubt, persalts can be incorporated in the instant washing compositions, preferably in an amount of up to 30%, such as 1 to 20%, and sometimes in a weight ratio to the invention imido peroxyacid of from 5:1 to 1:5. A diluent commonly present in these washing compositions is sodium sulphate, often from 5 to 50%, because it also functions as a processing aid. The previously mentioned salts that enable a halogen to be generated in situ can likewise be present in the washing compositions, which can then enjoy the alternative name of sanitising compositions.

The washing compositions can contain a number of optional components, sometimes alternatively called auxiliary agents. These agents which can each individually be included include soil anti redeposition agents (SARDs), dye transfer inhibitors, optical brightening agents (OBAs), stabilisers, corrosion inhibitors, bactericides, dyes, perfumes, foam enhancers, foam inhibitors, pH regulators and absorbents. The amount for each auxiliary agent is often selected in the range of 0.02 to 0.2% for dyes and perfumes and from 0.1 to 2% for each of the other auxiliary agents. It is preferable to select auxiliary agents which are known not to interact with peroxygen compounds during storage or to coat the agent with or incorporate the agent in a known fashion within a matrix of a dispersible material such as a wax or the many other film-forming substances proposed in the literature for separating organic peroxygen compounds from co-components, eg in EP-B-00 27 693 to Interlox Chemicals Limited. Such substances can also function as granulating aids (binders), if the invention compositions

are granulated or agglomerated. Examples of suitable SARDs include carboxymethyl cellulose particularly the sodium salt, polyvinylpyrrolidone and examples of OBAs include derivatives of diaminostilbene sulphonic acid and 1,3-  
5 diaryl-2-pyrazolines and aminocoumarins.

The invention washing compositions can be dampened or dissolved in a little water for cleaning and disinfecting non-adsorbent surfaces such as walls, floors, work surfaces, vessels, baths, sinks and sanitary-ware of metal, plastics,  
10 ceramics or glass, wood and rubber.

One of the main intended uses of the washing compositions is to cleanse and indeed also disinfect soiled adsorbent materials such as household laundry items or other articles made especially from cotton, rayon, flax or wool or  
15 man-made fibres such as polyesters or polyamides. The cleansing processes can be carried out at ambient temperature or at elevated temperature up to the boiling temperature of the washing solution. The more preferred washing temperature for laundry is from 30 to 60°C. In  
20 laundering, it is desirable to introduce sufficient washing composition and/or bleach additive composition to provide at least 5ppm avox from the imido peroxyacid, and often from 10 to 50ppm avox, ppm indicating parts per million by weight and avox indicating available oxygen. This can often  
25 be provided by the introduction of the invention washing composition selected in the range of 1 to 25gpl, or bleach additive composition selected in the range of from 0.5 to 10 gpl, the selection taking into account the concentration of imido peroxyacid therein. The presence of persalts in  
30 the wash can supplement avox levels, for example by amounts of from 10 to 100 ppm avox. In use, depending upon whether and the extent to which alkaline materials, especially builders, are present in the composition itself or in any accompanying washing composition, the compositions generate  
35 upon dissolution either a mildly acidic through to especially a mildly alkaline pH. It is preferred to generate a pH of from 7.5 to 9.5 and especially around pH of

8 to about 9.0 to optimise bleaching/washing performance from the peroxyacid.

For use in disinfection, it is often preferable to employ an invention peroxyacid concentration of up to 200ppm avox and in many instances from 25 to 100 ppm avox. It is also suitable to employ a solution spanning neutrality, from mildly acidic, such as at least pH 4 up to mildly alkaline, such as pH 9. In order to attain a pH in such a range, the choice of builders/diluents is so made as to avoid highly alkaline materials and instead select those that generate mild acidity or alkalinity such as sodium dihydrogen phosphate.

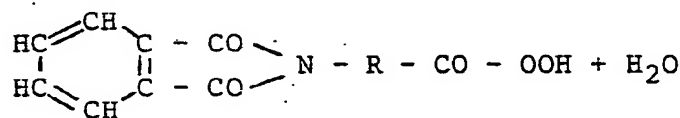
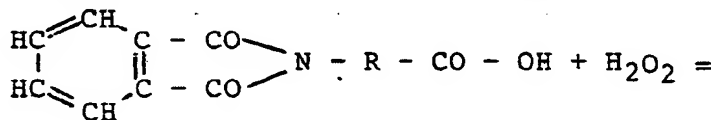
The washing processes for laundry can be carried out in currently available equipment. Washing times typically range from about 10 minutes to 30 minutes. Hand washing and extended steeping using solutions of the invention compositions can alternatively or additionally be used. Specialist variations of the invention compositions, such as those intended for nappy sanitisation/cleansing or for denture cleansing are preferably used in the accepted manner for prior art compositions, for example steeping a soiled nappy in a warm peracid-containing solution for several hours before washing it using laundry techniques.

Having described the invention in general terms, specific embodiments will now be described more fully by way of example only.

Example 1 and Comparison C2.

Preparation of imido peroxyacids.

In each Example and Comparison, the reaction equation for the acid catalysed reaction was



The general preparative route adopted for the first preparation of each peroxyacid was as follows:-

An imido carboxylic acid starting material for TIP6 was prepared by condensing phthalic anhydride with 6-amino-  
5 aminohexanoic acid. The results from IR and NMR analyses confirmed the presence of phthalimide and carboxylic acid moieties, and acid titration confirmed that there was only one acid group per molecule. The measured melting point of phthalimido-6-hexanoic acid 105°C, lit 107-8°C is also  
10 confirmatory that the preparation yields the specified starting materials. The starting material for TIP2 was obtained commercially.

#### Example 1

A weighed amount of an imidoaryl carboxylic acid (5g)  
15 was introduced into stirred methanesulphonic acid (25mls) in a beaker, forming a solution or suspension depending upon the solubility of the reactant, and the mixture was cooled to 3°C in a water/ice bath. Hydrogen peroxide assaying 85% w/w approx. aqueous solution, was pumped via a peristaltic  
20 pump with continued stirring into the reaction mixture progressively during a period of about 5 to 10 minutes at a rate controlled so that the mixture's temperature did not rise above 5°C, until a total amount of 3.5 moles per mole of carboxylic acid had been introduced, ie a 2.5 molar  
25 excess compared with the stoichiometric amount. The reaction mixture was then kept at about 6°C for a further 50 minutes. By the end of the reaction a substantial fraction of the carboxylic acid had been oxidised to the corresponding peroxy-carboxylic acid, which precipitated out  
30 of solution.

The reaction mixture was poured into about 3 times as much iced water per volume of reaction mixture, filtered and the filter cake washed twice with about 30 to 40 mls of cool water each time and finally air-dried.

35 The yield of solid was 5.0g having an avox content of 5.72 which indicates a purity of 99% (theoretical avox 5.76%) and a "melting" point of the pure product of 92°C.

The peroxyacid product is phthalimido-6-peroxyhexanoic acid (TIP6).

The comparison product phthalimido-2-peroxyacetic acid (TIP2) was made by the same reaction process, but modified by employing a 10g imido carboxylic acid scale and a 2 hours post-addition stirring of the reaction mixture. The white product obtained had an avox of 7.22% compared with a theoretical avox of 7.24%, indicating >99% purity.

The avox was measured by a standard technique in which a measured weight of sample was dissolved in acetic acid, if necessary augmented with dichloromethane to ensure that the sample is completely dissolved. The sample is then contacted with a measured amount of sodium carbonate stabilised sodium iodide, in the presence of ferric chloride, allowed to react for 10 minutes in the dark, and the resultant solution is titrated against standardised sodium thiosulphate solution until the pale yellow coloured solution becomes colourless. The result is compared with a corresponding titration against a blank solution, and from the difference the avox is calculated.

Both of the isolated peroxyacid products were analysed by conventional IR and TIP6 by NMR techniques to confirm the presence of imido and percarboxylic acid groups in the product molecule.

For the product of Ex 1, TIP6, three significant infra-red peaks were found. Two peaks were observed with centres at 1770 and 1710  $\text{cm}^{-1}$ , which correspond to a five membered imide ring. The third peak was observed having a centre at 1760  $\text{cm}^{-1}$ , indicative of carbonyl stretching in a peroxycarboxylic acid which is a substituent of an alkylene chain. The spectrum of TIP2 contained similar peaks, at respectively 1780 and 1710  $\text{cm}^{-1}$  and 1765  $\text{cm}^{-1}$ . It was also observed for both products that there was a substantial absence of peaks at about 3360  $\text{cm}^{-1}$ , or in the regions of 1640 to 1675  $\text{cm}^{-1}$ , and 1530 to 1545  $\text{cm}^{-1}$ , which would have indicated the presence of an amide group obtained by opening of the imide ring during the peroxidation reaction. The

spectra for the corresponding imidocarboxylic acid starting materials contained two significant peaks, a sharp peak at  $1770\text{ cm}^{-1}$  and a broad peak at about  $1720\text{ cm}^{-1}$ , which correspond to the two peaks for the imide structure, but the second one merging with a similarly located carboxylic acid peak. Thus, for the IR data 4, it can be deduced that the product retained its phthalimido structure and gained a percarboxylic acid group.

TIP6 was also analysed by proton NMR in  $\text{CD}_2\text{Cl}_2$  and the corresponding carboxylic acid likewise analysed, but in  $\text{CDCl}_3$ . The characteristics peaks for the two compounds, together with an indication whether they are singlet (s), triplet (t) or multiple (m), are listed in Table 1 below. In the column attributing the shift to particular H atoms, a distinction is drawn between the intended and adjacent methylene groups by specifying the former as  $-\text{CH}_2$  and the latter as Me.

Table 1

Chemical shifts for			
20	Carboxylic acid	TIP6	Peak attributed to H in
	11.4 s		$-\text{CO}-\text{OH}$
		12-9.5 broad	$-\text{CO}-\text{OOH}$
	7.7 m	7.8 m	4 x benzene C-H
	3.6 t	3.7 t	2 x N- $\text{CH}_2$
25	2.3 t	2.45 t	2 x CO- $\text{CH}_2$
	1.6 m	1.75 m	4 x N-Me- $\text{CH}_2$ or CO-Me- $\text{CH}_2$
	1.3 t	1.4 t	2 x Me- $\text{CH}_2$ -Me

It will be recognised that the NMR data given in Table 1 confirms the structures implicit from the reaction equations, the avox test and the IR scan.

### Example 3

#### Alternative preparation of TIP6

In this Example, a solution of Caro's acid was prepared by mixing sulphuric acid (98% w/w, 40g) and hydrogen peroxide solution (85% w/w, 5.3g) and demineralised water (11.5g) with cooling to  $12^\circ\text{C}$ , thereby obtaining an equilibrium mixture. The mixture was cooled to  $4^\circ\text{C}$ .



Phthalimido-6-hexanoic acid (10g) was then introduced, resulting after 10 minutes stirring in a slurry which was stirred, still at 20°C for a further 30 minutes. The reaction mixture was then quenched with ice (100g), and  
5 filtered. The white precipitate was washed with cold water (3 x 100ml) allowed to dry and then analysed. The yield of product was 10.1g of product having an avox of 5.43%.

The peroxyacids were subjected to a number of tests to determine their effectiveness as a bleach, their hazard  
10 rating and their storage stability. The compounds were also compared in these tests with a reference peroxyacid, diperoxydodecanedioic acid, DPDDA, a peroxyacid that has emerged during the last eight years as a favoured organic peroxide amongst washing composition manufacturers like  
15 Procter & Gamble.

The tests were carried out as follows:-

Storage stability

In this test, weighed samples of the peroxyacid are individually sealed in glass phials with a bubbler cap that  
20 permits excess internal pressure to vent to atmosphere, and stored in a dark chamber that is thermostatically controlled to 32°C. The avox of the peroxyacid is measured shortly after its preparation ie  $A_0$  and after predetermined storage intervals,  $A_S$ , the measurement being made on entire  
25 individual samples. The stability results of stored samples are  $A_S/A_0$ , quoted as a percentage, the higher the better.

Avox is measured using the same method as described hereinabove.

It will be recognised that the storage stability of the  
30 peroxyacid by itself is an extremely important characteristic of a peroxyacid, not only because the compound is likely to be stored by itself before it is incorporated in specific compositions, but also because represents the intrinsic stability of the compound, the  
35 maximum attainable if the remaining components of compositions containing it are benign.

A + indicates that the compound is according to the

invention whereas a - indicates that it is present by way of comparison.

Table 2

Compound	Proportion of avox remaining after		
	1 week	4 weeks	longest/ n weeks
5 + TIP6	97	99	88/ 8w
- TIP2	100	87	13/ 16w
- DPDDA	97	85	

From Table 2, it can be seen that TIP6 had very good inherent storage stability, but that the comparison TIP2 was quite stable initially but after a month began to lose its available oxygen much more rapidly.

The improvement in peroxyacid stability for TIP6 over TIP2 is believed to be attributable to the number of methylene groups that separate the percarboxylic acid moiety from the imide ring.

#### Hazard Rating

Two tests are described below to demonstrate the hazard rating of the peroxyacid. They are respectively an impact sensitivity test and a pressure-time test.

In the impact sensitivity test, a weight (in kg) is dropped once from a measured height (in cm) onto a fresh sample of the peroxyacid held in the anvil. The sample is thus subjected to an impact, normally expressed as kg-cm (1kg-cm =  $9.8 \times 10^{-2}$ J) that is proportionate to the height and weight. The test is carried out many times at each impact strength, and is observed to see whether the sample responds, by charring, emitting smoke or at worst undergoing a minor explosion. The tests start at a low impact strength and are continued at increasing strenghts until the limiting result is obtained, being the earlier of either 50% of the tests at that impact strength give positive results or a figure of 500kg-cm is reached, which past experience indicates to represent a non-impact-sensitive product. The limiting result in kg-cm is shown in Tables summarising the results, the higher the better.

In the pressure-time test measuring response to thermal

shock, 2g samples of the test material is placed inside an 18ml steel bomb, and its decomposition initiated. The consequential rise in pressure is monitored and plotted or displayed against elapsed time, expressed in milliseconds.

- 5 In Table 3, the time is given for the pressure in the bomb generated by the sample to increase from 100 to 300 psi, ie from  $6.895 \times 10^5$  Pa to  $2.068 \times 10^6$  Pa, the longer the better. The symbol oo indicates that a pressure of 300 psi was not reached, ie a period of infinite duration. By way  
10 of interpretation, a time of less than 30 milliseconds indicates that the material is potentially explosive, a time of 30 to 60 milliseconds indicates that it is marginally explosive, and to allow a safety margin, it is preferred to be around 100 milliseconds or longer.

15

Table 3

Compound	Hazards rating results	
	Impact	p-t
	kg-cm	msec
+ TIP6	>500	oo
20 - TIP2	75	30
- DPDDA	>500	30

- From Table 3, it can be seen that the invention imido-peroxyacid TIP6 was much safer as demonstrated by the impact test than the comparison imido peroxyacid TIP2, and in the  
25 pressure-time test, it was significantly safer than both the reference compound DPDDA and TIP2. These tests show that the selection of a suitable alkanolic acid substituent for the imide group is of crucial significance in order to obtain a product that is inherently safe to handle.

### 30 Bleach/washing evaluation

- The effectiveness of the invention and comparison peroxyacids was tested by washing swatches of cotton cloth that had been preimpregnated in a standard manner with one of four representative stains, tea, red wine, grass and blue  
35 polish. The evaluations were carried out in a laboratory scale washing machine, a "Tergotometer" (Trade Mark) available from the US Testing Corporation, under identical

standardised conditions. The washing solution comprised local Cheshire tap water, hardness of about 160 to 180 ppm hardness as calcium carbonate, in which was dissolved a peroxyacid-free washing composition at 6.5 g/l. Base detergent compositions DBPF used in the trials summarised in table 4 and DBNSP used in the trials summarised in Table 4A had the respective approximate analyses:-

Composition		DBPF	DBNSP
Component		% w/w	% w/w
10	Anionic surfactant	9	9
	Nonionic surfactant	6	8
	Other organics	17	1
	Sodium carbonate	<1	3
	Sodium sulphate	25	19
15	Sodium phosphate (as anhydrous)	<1	36
	Sodium silicate	8	10
	Zeolite A	23	-
	Sodium borate		4
	Water	balance	

20 A weighed amount of peroxyacid was introduced into the washing solution to provide a peracid avox of 25 ppm therein, assuming total dissolution. This corresponds to a molar concentration of  $1.56 \times 10^{-3} \text{M}$  monoperoxyacid. The washing solution was kept at pH9 and at 40°C during the washing period of 20 minutes. The swatches were then rinsed and dried and the extent of stain removal was determined by comparing the reflectance of the washed cloth,  $R_w$ , with that of the pre-washed, stained cloth,  $R_s$ , and that of the unstained cloth,  $R_u$ . The measurements were obtained using an Instrumental Colour System "Micromatch" (Trade Mark) reflectance spectrophotometer equipped with a Xenon lamp filtered through a D65 conversion filter to approximate to CIE artificial daylight. Stain Removal, expressed as a percentage, was calculated using the formula:-

$$35 \quad \%SR = 100 \times [R_w - R_s] / [R_u - R_s]$$

It will be recognised that by demonstrating the washing capability of the peroxyacids in this way, the tests using

the invention peroxyacids are in themselves Examples of washing processes according to other aspects of the present invention. Similarly, since the swatches had not been stored in sterile conditions before being washed, the washing procedure will act simultaneously to disinfect them.

The results quoted below are the mean of two evaluations. Comparative results on the same stained cloths using the washing composition by itself, ie without any added peracid, are designated "base".

10

Table 4

Ex/Comp No	Peracid employed	% Stain Removal				
		Red Wine	Grass	Tea	Blue Polish	Average Removal
C4	base	47	57	32	43	45
15 5	TIP6	74	80	71	52	69
C6	DPDDA	59	70	56	53	60

From Table 4, it can be seen that the invention peracid are very effective bleaching agent at hand-hot washing temperatures, not only by comparison with a peracid-free base composition, but also when measured against DPDDA.

In order to further illustrate the effectiveness of the invention peroxyacid as a bleach in a washing process, in Ex5A, the washing procedure for Example 5 was repeated in the same apparatus and using the same washing conditions in respect of the four representative stains named above, but in a solution of DBNSP rather than DBPF. The same trials were conducted using peracid-free solution, C4A, and solutions containing the related phthalimidoperoxyacids, TIP2, identified above, TIP3 (phthalimidoperpropanoic acid) and TIP4 (phthalimido-perbutanoic acid) at the same molar concentration of  $1.53 \times 10^{-3}$  M in respectively C4B, C4C and C4D. The stain removals for each stain were measured as before and the average results are given in Table 4A below, together with the increase in average stain removal over the detergent solution alone.

25

Table 4A

Ex/Comp	Peracid	% Stain Removal Average of 4 stains	% increase in Stain Removal
C4A	base	60	-
5 C4B	TIP2	72	12
C4C	TIP3	78	18
C4D	TIP4	78	18
Ex5A	TIP6	82	22

From Table 4A, it can be seen that the invention  
 10 peroxyacid TIP6 performed significantly better at stain  
 removal than the shorter chain peroxyacids in the same class  
 of compounds when measured against the range of  
 representative stains. The results in Table 4A show that  
 TIP6 has a more advantageous balance of hydrophobicity and  
 15 hydrophilicity than does TIP2, TIP3 or TIP4, thereby  
 enabling it to treat overall the range of hydrophilic and  
 hydrophobic stains more effectively.

The foregoing tests demonstrate that the invention  
 peroxyacid is superior to the best of the peroxyacids tested  
 20 and described in EP-A-0 325 288 and EP-A-0 325 289 by  
 Ausimont S.r.l. and published between the priority date and  
 the application date for the present application.

#### Bleach Additive Formulations

25 Representative formulations are made by dry mixing  
 particulate invention peroxyacid with a premixture of the  
 remaining components. The peracids have the names given in  
 Tables 1 and 2. LAS represents a linear alkyl benzene  
 sulphonate, sodium salt, average alkyl length of C11.5, and  
 30 OBA represents an optical brightening agent.

Table 5

Example No	7	8	9
Particulate Components	% w/w	% w/w	% w/w
TIP6 (5.7% avox)	7.2	29	58
35 LAS	3	4	5
OBA + chelate	0.2	0.2	0.2
Sodium sulphate	89.6	66.8	36.8

Dosing of formulations 7, 8 and 9 each at 1.25 gpl provides respectively avox concentrations in solution of approximately 5ppm, 20ppm and 40ppm. Solid bleach additive compositions containing a pH buffer to lower the solution pH closer to about pH 8.5, and hence improve stain removal are made by replacing about 10% w/w of the sodium sulphate by boric acid.

#### Washing Formulations

Representative washing compositions according to the present invention are made by dry mixing the particulate invention peroxyacid with a blend of the other components shown in Table 6. The abbreviations STPP and PBS1 represent respectively sodium tripolyphosphate and sodium perborate monohydrate. The chelating agent is EDTMP, ethylene diamino (tetramethylene phosphonate), Na salt.

Table 6

Example No	10	11	12	13	14	15
Components	% w/w	% w/w	% w/w	% w/w	% w/w	% w/w
20 TIP6 (5.7% avox)	2.2	4.3	6.4	3.2	5.4	7.6
LAS	7.0	9.6	8.6	7.0	6.0	6.0
Alcohol Ethoxylate	5.1	3.8	5.7	2.5	6.0	7.0
STPP	34.0	26.1		40.0	30.0	30.0
Zeolite A			22.5			
25 Carboxylate builder		2.0	15.0			
Sodium sulphate	13.8	37.0	23.0	18.0	23.0	15.3
Sodium silicate	14.0	6.7	7.6	6.5	5.0	5.0
Soap	6.5			3.0	3.0	2.0
Buffer (boric acid)	10.0			10.0	10.0	10.0
30 PBS1						9.0
CMC	1.0	1.0	1.0	1.0	1.0	1.0
Minors (Chelate + OBA + Perfume etc)	0.4	0.4	0.4	0.6	0.3	0.5
Water				balance		

Use of example formulations 10 to 15 at a concentration of 8 gpl in the washing liquor, a typical level for front loading washing machines in Europe, results in peracid avox

27

concentrations of approximately 10, 20, 30, 15, 25, and 35 ppm respectively.

### Sanitizer Formulations

- 5 Representative formulations are made by dry mixing the specified invention peroxyacids with the other components specified in Table 7.

Table 7

Example No	16	17	18
10 Particulate Components	% w/w	% w/w	% w/w
TIP6 (5.7% avox)	5.2	8.6	12.4
LAS	9.0	7.0	5.0
Sodium carbonate	20.0	23.0	
STPP	10.0	10.0	10.0
15 Sodium bicarbonate			26.0
Sodium chloride	47.3	50.7	45.8
Borax	8.5		
Organic chelate		1.2	0.8

- 20 When these formulations are dosed into a nappy (or similar article) sanitising solution in an amount of 5 gpl, the invention peroxyacids provide an avox of respectively 35, 15 and 25 ppm approximately.

### Dilute Disinfectant Compositions

- 25 Particulate disinfectant compositions are made by dry mixing the components specified in Table 8.

Table 8

Example No	19	20	21
Particulate Components	% w/w	% w/w	% w/w
30 TIP6 (5.7% avox)	4.9	8.9	12.3
Sodium dihydrogen phosphate	10.0	10.0	10.0
Boric acid	5.0	5.0	5.0
Corrosion Inhibitor	1.0	1.0	1.0
35 Perfume	0.5	0.5	0.5
Sodium sulphate	78.6	75.2	71.2



When these formulations are employed at a dose level of 1 g/lp in an aqueous medium requiring disinfection, the concentration of avox therein is respectively 3, 5 and 7 ppm.

5

Disintegrating Tablet compositions - suitable for dentures

Representative compositions of this type are made by dry mixing the components given in Table 9 below, and then subjecting them to compression in the mould of a tableting machine to make tablet weighing about 4g. The polyethyleneglycol binder av mol weight 6000 is designated PEG 6000, the disintegrant was a cross linked polyvinylpyrrolidone available under the Trademark POLYPLASDONE XL and the lubricant was sodium lauryl sulphate.

15

Table 9

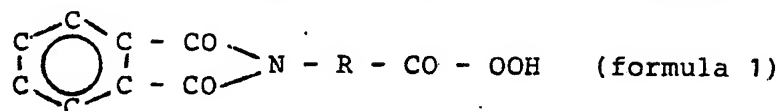
Example No	22	23	24
Particulate Components	% w/w	% w/w	% w/w
TIP6 (5.7% avox)	7.3	9.4	11.1
20 Succinic acid	25.2	15.0	15.0
Sodium Bicarbonate		25.5	40.0
Sodium Carbonate	10.0		
PEG 6000 (binder)	6.0	6.0	6.0
PVP disintegrant	1.0	1.0	1.0
25 Lubricant	0.2	0.2	0.2
Sodium sulphate	50.3	42.9	26.7

When one tablet of composition 22, 23 or 24 is introduced into water it generates respectively 17, 21.5 or 25.6 mg avox.

29

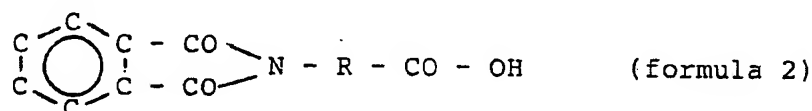
Claims:-

1. An organic peroxyacid which satisfies general formula (1):-



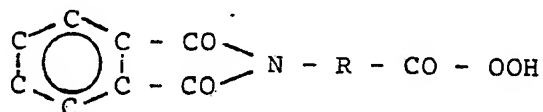
in which R represents a pentamethylene group.

2. A process for the production of organic peroxyacids of formula (1) in which an organic carboxylic acid of formula (2)



in which R represents a pentamethylene group is reacted with excess aqueous hydrogen peroxide in a strong acid medium selected from organic sulphonic acids and or mineral acids at a reaction temperature of from about -5 to 50°C until at least some percarboxylic acid product has been produced as a solid, and thereafter recovering the solid product from the reaction mixture.

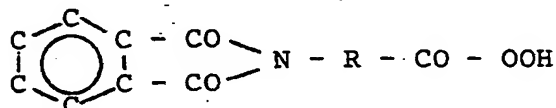
3. A process according to claim 2 in which the organic sulphonic acid reaction medium is methane sulphonic acid.
4. A process according to claim 2 in which sulphuric acid is employed as a premixture with the hydrogen peroxide reactant which thereby contains peroxomonosulphuric acid.
5. A bleach composition containing from 1 to 80% w/w of an organic peroxyacid which satisfies the general formulae (1)



30

in which R represents a pentamethylene group and from 99 to 20% w/w of a diluent.

6. A washing composition containing from 0.5 to 50% w/w of an organic peroxyacid which satisfies the general formulae (1)



in which R represents a pentamethylene group and from 1 to 90% surfactant, from 0 to 90% detergent builder, from 0 to 90% diluent and from 0 to 20% minor components.

7. Use of a peroxyacid according to claim 1 or a composition according to claim 5 for bleaching and/or disinfection.
8. Use of a peroxyacid according to claim 1 or a composition according to claim 6 for washing articles.

# INTERNATIONAL SEARCH REPORT

International Application No. PCT/GB 89/01524

## I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) \*

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC5: C 07 D 209/48, C 11 D 3/39, D 06 L 3/02

## II. FIELDS SEARCHED

Minimum Documentation Searched ?

Classification System (

Classification Symbols

IPC5 : C 07 D; C 11 D; D 06 L

Documentation Searched other than Minimum Documentation  
to the Extent that such Documents are included in the Fields Searched \*

## III. DOCUMENTS CONSIDERED TO BE RELEVANT \*

Category \* | Citation of Document, \*\* with indication, where appropriate, of the relevant passages \*\* | Relevant to Claim No. \*\*

X,P	EP, A1, 0325288 (AUSIMONT S.R.L.) 26 July 1989, cited in the description, see especially page 2	1-8
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X,P	EP, A1, 0325289 (AUSIMONT S.R.L.) 26 July 1989, cited in the description, see the whole document	1-8
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A	EP, A2, 0170386 (THE PROCTER & GAMBLE COMPANY) 5 February 1986, see the whole document	1-8
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\* Special categories of cited documents: \*\*

"A" document defining the general state of the art which is not  
considered to be of particular relevance

"E" earlier document but published on or after the international  
filing date

"L" document which may throw doubts on priority claim(s) or  
which is cited to establish the publication date of another  
citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or  
other means

"P" document published prior to the international filing date but  
later than the priority date claimed

"T" later document published after the international filing date  
or priority date and not in conflict with the application but  
cited to understand the principle or theory underlying the  
invention

"X" document of particular relevance; the claimed invention  
cannot be considered novel or cannot be considered to  
involve an inventive step

"Y" document of particular relevance; the claimed invention  
cannot be considered to involve an inventive step when the  
document is combined with one or more other such docu-  
ments, such combination being obvious to a person skilled  
in the art.

"Z" document member of the same patent family

## IV. CERTIFICATION

Date of the Actual Completion of the International Search

21st March 1990

Date of Mailing of this International Search Report

10. 04. 90

International Searching Authority

EUROPEAN PATENT OFFICE

Signature of Authorized Officer

L. ROSSI

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
A	Chemical Abstracts, volume 57, 1962, (Columbus, Ohio, US), K. Balenovic et al: "Preparation of some peroxy acids derived from optically active amino acids", column 15224i-15225c, & J. Chem. Soc. 1962, 3821-2.	1-4
A	Chemical Abstracts, volume 56, 1962, (Columbus, Ohio, US), K. Balenovic et al: "Asymmetric synthesis of sulfoxides with -substituted monoperglutaric acids", column 4663 e-h, & Chem. & Ind. (London) 1961, 469-70.	1

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